2.8

LWG
LOWER WILLAMETTE GROUP

FISH TISSUE HOMOGENIZATION AND SHIPPING SOP

ROUND 1
PORTLAND HARBOR RI/FS

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Prepared By:

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INTRODUCTION

Homogenization and shipment of fish tissue samples collected to support the Portland Harbor RI/FS were previously described in the Round 1A Field Sampling Plan (FSP) (SEA et al. 2002a), and the Round 1 FSP (SEA 2002). This standard operating procedure (SOP) modifies and clarifies the procedures previously presented. The procedures described in this SOP are in accordance with U.S. Environmental Protection Agency (EPA) guidance (EPA 2000).

Separate SOPs for the fish tissue sample collection and sample compositing and shipment (pre-homogenized samples) for the Portland Harbor RI/FS have been provided to EPA under separate cover (SEA et al. 2002b, SEA et al. 2002c).

SAMPLE RECEIPT

All tissue samples will be shipped to Axys Analytical Services, Ltd. (Sidney, BC, Canada) for tissue sample homogenization as described in the Portland Harbor RI/FS Sample Composting and Shipment SOP (SEA et al. 2002c).

All samples arriving at Axys laboratory will be processed according to Axys sample receiving SOP (Axys 2002a and b). This will also include generation of email to the Chemistry QA Manager upon receipt and check in of the samples.

Each cooler arriving at Axys will have a unique identifier on it and will be processed individually. All other coolers will be placed into a secure area and await individual login. The outside of the cooler will be inspected for shipment damage and all custody seals inspected for damage. Once the cooler has been inspected the login procedures will start. Intact custody seals will be removed using a razor blade. These tags will be placed onto a clean piece of paper and processed as evidence that the cooler arrived under custody seal. Once the seals have been removed the cooler will be opened. The contents of the cooler will be inspected for leakage, possible cross contamination, and temperature. Results of this inspection will be noted in the sample receipt log (Figure 1). The original COC and FEDEX commercial weigh bill, which acted as the COC during shipment by FEDEX, will be removed and all the information transferred into the laboratory information management system (LIMS) and the sample receipt log. The commercial invoice (Figure 2) will be placed into that cooler's individual paperwork folder.

Samples will be removed from the cooler and placed on a clean surface. No other samples will be checked in during the time that the LWG's samples are being cross-checked against the COC. Samples should be bound together as composites as described by SEA et al (2002c). If for some reason there are individual samples, these samples will be placed on hold and the Chemistry QA Manager notified.

Sample tags will be cross-checked against the COC and any discrepancies noted on the corrective action form. Samples with COC discrepancies will also be placed on hold and the Chemistry QA Manager notified. Once the contents of the cooler have been verified, the samples will be logged into the laboratory LIMS (Figure 3) and any non-conformances will be discussed with the Chemistry QA Manager (this can be done once entire shipment has been logged into LIMS).

The LIMS will generate a work order for sample homogenization. Samples will be processed in compliance with Axys (2002c).

HOMOGENIZATION

Tissue homogenization will occur using Axys (2002c), with these project-specific stipulations:

- All tissue for the LWG will be place on aluminum foil dull side up, to avoid possible contamination associated with the shiny surface of the foil.
- Tissue will be processed on Teflon cutting surfaces or aluminum foil, not on paper towels as noted in Axys (2002c).
- Samples will be processed in the sample delivery group they arrived in.

It is recognized that scarring of Teflon boards might occur during tissue processing and any residues trapped in these scars represent a cross-contamination potential. Extra care will be taken in the decontamination process between samples to minimize this potential. It has been stated in the FSP that any liquors and fluid coming from the tissue will be processed along with the samples. Care will be taken to achieve this by wiping the aluminum foil that the tissue arrives in into the homogenate, and by transferring all liquors and liquid generated during the chopping process into the homogenate. These fluids will be homogenized as part of the tissue sample. It is also recognized that in certain cases the volume of tissue will be low. In these cases, the meat grinders will be *carefully* scraped to retain the maximum amount of tissue. Careful scraping is required to avoid the introduction of trace metal constituents from the grinders into the sample. If there is ample amount of tissue for the analysis, then the scraping step will be omitted. Also, when sample volume is low, the tissue will be passed through the grinder and then formed into log shaped piles that are placed in a bowl that sits atop dry ice to be re-chilled prior to the second grinding. For sub-yearling Chinook salmon two grindings will be sufficient.

SUB-SAMPLING FOR ANALYSIS

Homogenized tissue samples will be divided into multiple jars for analysis, QA/re-analysis (as needed), and archiving. This is a critical step in the COC and Axys laboratory personnel will keep close records of the composited samples they are homogenizing using Table 1 and Figure 4. Table 1 lists the number of jars and appropriate jar size for the analyses. This table also describes how many containers and which lab will be receiving the samples.

During the homogenization process various weights of tissue are recorded (Axys 2002c). This will include the weight of individual fish or fish fillets that go into each composite sample before being placed in the grinder. All weights will be recorded to the nearest 0.1g on the sample processing record. Figure 4 is a tracking log for homogenate weight and cooler packaging information. This log will be kept and a copy of each page pertaining to a specific cooler will be copied and travel with the cooler paperwork (if the laboratory generates forms of a similar nature they may used). On the outside of the jar, either on the label or on the jar lid, a numbering scheme for the amount of jars one sample produces (i.e., 1 of 8, 2 of 8, etc.) will be noted. Axys will also note the amount (in grams) of tissue in each jar to be sent to Columbia Analytical Services (CAS).

It is understood that there will likely be some species that will not yield the desired amount of tissue. In these instances, smaller jars will be used to supply more opportunity for re-extraction without multiple thawings. Conversely, there will also be samples with an abundance of tissue. In these cases, a reserve container of 500g will be archived at Axys.

SHIPPING SAMPLES

Homogenized samples will be shipped from Axys to CAS for analysis when there is a sufficient volume of samples to fill a cooler, or as required due to holding time constraints. The following steps will be followed when each cooler is shipped:

- Samples will be completely frozen prior to shipping (-20°C)
- Coolers will be medium sized (24"x14"x15"). They will be decontaminated/cleaned and identified with a cooler number
- Dry ice will line the bottom of the cooler
- Samples will be removed from the freezer, checked off the freezer log, jars will be wrapped in bubble wrap, and placed into a cooler
- The sample information will be noted on a new COC
- The top layer will be dry ice

- The weight of the cooler will not exceed 50 lbs for health and safety reasons
- A temperature blank will be added (this will be frozen as if it were a sample)
- Paperwork will be prepared (shipment, COC, and placards)
- New COC and copies of the original paperwork will be placed into a Ziploc bag and taped to the inside of the cooler lid
- Placards will be taped to the cooler that state:
  - ↑ THIS SIDE UP ↑
  - Environmental Samples of No Commercial Value
  - In case of delay or emergency please contact Janet Cloutier 360-705-3534 or Ian Stupakoff 206-419-0809
  - Fragile, Handle with Care
  - Samples Must Remain Frozen
- Each cooler will be custody sealed on the three sides that open, taped around the girth and width, and placed into a freezer for holding until picked up by FEDEX.

Shipping by FEDEX is the preferred method from Axys to CAS in Kelso, WA. The coolers will be delivered to the local FEDEX and processed with international shipping papers. These contain a FEDEX weigh bill and a Commercial Invoice. The FEDEX weigh bill will act as an extension of the COC. The persons shipping the coolers will retain a copy of the weighbill and either email or fax a copy of the weigh bill (that contains the tracking number) to the Chemistry QA manager upon shipping. The original will be provided to SEA for incorporation in the final data package. Samples will be shipped in the middle of the week to ensure that weekend delays are avoided.

It is critical that the samples be shipped and received frozen ($\leq 0°C$). Samples will be packed and shipped on dry ice. This will ensure that samples will arrive below -20°C. If a cooler arrives > 0°C but $\leq 4°C$, it will be assumed that hold time will start from the time it was packaged. All labs are to notify the chemistry QA manager upon receipt of such non-conforming samples immediately. Upon notification, the chemistry QA manager will notify the field QA manager and the CERCLA coordinator. Finally, if a cooler arrives > 4°C, the same notification process will ensue. The CERCLA project coordinator will consult with the EPA QA manager prior to making a decision about analyzing such samples.

Effective communication between the laboratories and the Chemistry QA Manager is critical in this project. Key project staff that will maintain contact during key project coordination periods, e.g., when samples are being shipped from Axys to CAS, are:

Janet Cloutier - LWG Chemistry QA Manager, Striplin Environmental Associates, Olympia, WA: jcloutier@striplin.com 360-705-3534

Dr. Coreen Hamilton - PM Axys Laboratory, Sydney, BC: chamilton@axys.com 250-655-5800

Abbie Spielman - PM CAS Laboratory, Kelso, WA: aspielman@kelso.caslab.com 360-577-7222.

REFERENCES

Axys 2002a, Axys Analytical Services Ltd. Standard Operating Procedure: Logging-In Samples SOP# LAB-3 Rev No. 9 May 31, 2001.

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U.S. EPA. (Environmental Protection Agency). 2000 Guidance for assessing chemical contaminant data for use in fish advisories, Volume 1 - Fish sampling and analysis. Third Edition. EPA 823-B-00-007. U.S. Environmental Protection Agency, Office of Water, Washington, DC.

Figure 1 SAMPLE RECEIVING RECORD **AXYS Analytical Services Ltd**

SAMPLE RECEIVING RECORD

Waybill · Present _____ Absent _____		Waybill #·
Date Shipped.	Date Received:	Time Received.
Received By (print)	Signature:	
Axys Client and Contract #	Client Reference #:	

Condition of Shipping Container·

Temperature of Shipping Container on Receipt

Custody Seals: Absent : _____
Present :___ Intact ___ Broken ___
On: Shipping Container ___ Sample Container·___

Custody Seal Numbers:

Axys Sample IDs·

Log-in by (print): Signature:

Chain of Custody or Documents: Present· ____ Absent: ____ Traffic Report/Packing List: Y / N

Sample IDs	Y / N	Sample Tag Numbers	Y / N	
Location	Y / N	Sample Type	Y/ N	
Date & Time of Collection	Y / N	Preservative Added	Y / N	_____ (details)
Collector's Name	Y / N	Preservation Requested	Y / N	_____ (details)

Sample Acceptance Criteria Matrix Type :_____

Appropriate Container	Y / N	Correct Labelling	Y / N
Damaged Container	Y / N	Holding Time Exceeded	Y / N
Adequate Sample Size	Y / N	Appropriate Temperature	Y / N

Aqueous Samples: pH adjustment required Y / N Residual Cl required Y / N

Sample Tags: Present: ___ Absent: ___ **Sample Labels:** Present: _____ Absent _____

Sample Labels Cross Referenced to Chain of Custody	Y / N	Information Agrees	Y / N
Sample Tags Cross Referenced to Sample Labels	Y / N	Information Agrees	Y / N
Sample Tags Cross-Referenced to Chain of Custody	Y / N	Information Agrees	Y / N

Problems or Discrepancies

Action Taken

Figure 2. Commercial Invoice

COMMERCIAL INVOICE

DATE OF EXPORTATION	EXPORTER REFERENCE (i e , order no , invoice no , etc)
SHIPPER/EXPORTER (complete name and address)	CONSIGNEE (complete name and address
Country of Export	REASON FOR SHIPMENT
Country of Manufacture	
Country of Ultimate Destination	
International Air Waybill No	

MARKS / Nos	No of PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	Qty	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	TOTAL NO OF PKGS					TOTAL WEIGHT		TOTAL INVOICE VALUE

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN
DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT

SIGNATURE OF SHIPPER/EXPORTER (Type name and title, and sign) DATE

AXYS

Axys Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jul 03, 2002
11 31 AM

Login Number: L4822
Account: 4170 Striplin Environmental Associates 2002
Project:

AXYS ID	Client Sample Identification	Collect Date	Receive Date	PR	Due Date
L4822-1	1AFZ0203TSLSWBSOO		28-JUN-02		19-JUL-02
	LWG1AFZ0203TSLSWBSOO; Whole Body-Dry Rur				
WIF4, red cooler, floor, purposes	COC and sample label discrepancy, Samples on hold for PCB & DX only Homo and discarded for demonstration				
Tissue	HOLD				USD
Tissue	HOMOGENIZATION		1	Foil	CDN
Tissue	SUBSAMP1 W/O DATA				CDN
L4822-2	1AFZ0203TSLSFLCOO		28-JUN-02		19-JUL-02
	LWG1AFZ0203TSLSFLCOO; L. Side Fillet, skin on				
WIF4, red cooler, floor,	COC and sample label discrepancy; Samples on hold for PCB & DX only				
Tissue	HOLD				USD
Tissue	HOMOGENIZATION		1	Foil	CDN
Tissue	SUBSAMP1 W/O DATA				CDN
L4822-3	1AFZ0203TSLSFLCOO		28-JUN-02		19-JUL-02
	LWG1AFZ0203TSLSFLCOO; R. Side Fillet, Skinnec				
WIF4, red cooler, floor,	COC and sample label discrepancy, Samples on hold for PCB & DX only				
Tissue	HOLD				USD
Tissue	HOMOGENIZATION		1	Foil	CDN
Tissue	SUBSAMP1 W/O DATA				CDN
L4822-4	1AFZ0203TSSBFLCOO		28-JUN-02		19-JUL-02
	LWG1AFZ0203TSSBFLCOO; L. Side Fillet, Skin on				
WIF4, red cooler, floor;	COC and sample label discrepancy, Samples on hold for PCB & DX only				
Tissue	HOLD				USD
Tissue	HOMOGENIZATION		1	Foil	CDN
Tissue	SUBSAMP1 W/O DATA				CDN
L4822-5	1AFZ0203TSSBFLCOO		28-JUN-02		19-JUL-02
	LWG1AFZ0203TSSBFLCOO; R. Side Fillet, Skinne				
WIF4, red cooler, floor; purposes	COC and sample label discrepancy, Samples on hold for PCB & DX only Homo. and discarded for demonstration				
Tissue	HOLD				USD
Tissue	HOMOGENIZATION		1	Foil	CDN
Tissue	SUBSAMP1 W/O DATA				CDN
L4822-6	1A005FZ0809CCFLCOO		28-JUN-02		19-JUL-02
	LWG1A005FZ0809CCFLCOO; L. Side Fillet, Skin or				
WIF4, red cooler, floor,	COC and sample label discrepancy, Samples on hold for PCB & DX only				
Tissue	HOLD				USD
Tissue	HOMOGENIZATION		1	Foil	CDN
Tissue	SUBSAMP1 W/O DATA				CDN

Signature : _____

Date : _____

Axys Analytical Services Ltd.

Login Chain of Custody Report (ln01)

Jul. 03, 2002

11 31 AM

Login Number: L4822

Account: 4170 Striplin Environmental Associates 2002

Project:

AXYS ID	Client Sample Identification	Collect Date	Receive Date	PR	Due Date
L4822-7	1A005FZ0809CCFLCOO		28-JUN-02		19-JUL-02
			LWG1A005FZ0809CCFLCOO; R. Side Fillet, Skinn		
VIF4, red cooler, floor, COC and sample label discrepancy, Samples on hold for PCB & DX only					
Tissue	HOLD				USD
Tissue	HOMOGENIZATION		1 Foil		CDN
Tissue	SUBSAMP1 W/O DATA				CDN

Signature : _____

Date : _____

Figure 4 Sample Container Log

Sample ID	Sample	Jar Size	Weight in Jar	SDG	Cooler #	Date Packed in Cooler	Int
	___ of ___						
	___ of ___						
	___ of ___						
	___ of ___						
	___ of ___						
	___ of ___						
	___ of ___						
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	___ of ___						
	___ of ___						

Table 1. Number of Jars/Size per Analysis and Sample[3].

Species	Sample Type	CAS			Axys
		Metals + solids 8 oz sample + MS + Archive	PCB + PEST (lipids) + HERB 8 oz sample + MS + MSD + Archive	Hg[2] 4 oz sample + Dup + Archive	Diox/Furn + Cong 4 oz sample + MS + Archive
Crayfish	Whole body	3	4	3	3
Juvenile Chinook	Whole body	3	4	3	3
Lamprey ammocoetes	Whole body	3	4	3	3
Large scale sucker	Whole body	3	4	3	3
Peamouth	Whole body	3	4	3	3
Sculpin	Whole body	3	4	3	3
Northern pikeminnow	Whole body	3	4	3	3
Benthic invertebrates		3	4	3	3
Bullhead (Wallleye[1])	Whole body	3	4	3	3
Bullhead (Wallleye[1])	Fillet with skin	3	4		3
Bullhead (Wallleye[1])	Fillet without skin			3	
Black crappie	Whole body	3	4	3	3
Black crappie	Fillet with skin	3	4		3
Black crappie	Fillet without skin			3	
Smallmouth bass	Whole body	3	4	3	3
Smallmouth bass	Fillet with skin	3	4		3
Smallmouth bass	Fillet without skin			3	
Carp (Largescale sucker[1])	Whole body	3	4	3	3
Carp (Largescale sucker[1])	Fillet with skin	3	4		3
Carp (Largescale sucker[1])	Fillet without skin			3	

[1] = The species in parentheses may be collected as alternative species to those indicated

[2] = In the case of small sample volume use 2oz jars

[3] = Retain all tissue until Chemistry QA Manager is notified of the sample amount.